



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION VIII

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DENVER, COLORADO 80202-2466

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Ref: 8ES-MEB

November 6, 1995

MEMORANDUM

SUBJECT: Data validation for Rico Argentine Mine Site, Case #24008, SDG # MHDD72

FROM: Russ Leclerc
Chemist
Program Support Group, Technical Support Team

REC 11/6/95

TO: Greg Oberly
8HWM-SM

The Environmental Services Assistance Team (ESAT) has completed its review of data from the analysis of twenty soil samples for Contract Laboratory Program (CLP), Routine Analytical Services (RAS) total metals and cyanide analyses for **Rico Argentine Mine Site, Case 24008**, Sample Delivery Group (SDG) #MHDD72. I have evaluated ESAT's data validation package and agree with ESAT's review. Data in the enclosed package are acceptable with the qualifiers added to the data reports. Please refer to the attached ICF Kaiser data validation report including the narrative summary and comments for a full explanation of the data review findings.

If you have any questions, or if I can be of further assistance, please contact me at 312-6971.

Attachments



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REGION VIII
RAS INORGANIC - SUMMARY OF CLP DATA QUALITY ASSURANCE REVIEW

CASE	SITE NAME	SITE ID\OPERABLE UNIT
24008	Rico Argentine Mine	8ZZ/00
RPM NAME	ESAT TID - 08-9510-703	
Greg Oberly	ESAT WUD - 27	

LABORATORY	CONTRACT NO.	SDG	LABORATORY TPO/REGION
Southwest Laboratory of Oklahoma	68-D3-0040	MHDD72	Ray Flores/VI

DATA REVIEWER Kristy K. Grove REVIEW COMPLETION DATE 11/06/95

SAMPLE ID	SAMPLE LOCATION	MATRIX	DATE COLLECTED
MHCQ95	RA-SE-01	Soil	09/13/95
MHCQ96	RA-WSO-01	Soil	09/13/95
MHCQ97	RA-WSO-02	Soil	09/13/95
MHDD72	RA-SE-11	Soil	09/11/95
MHDD73	RA-SE-10	Soil	09/11/95
MHDD74	RA-WSO-06	Soil	09/11/95
MHDD75	RA-WSO-07	Soil	09/11/95
MHDD76	RA-WSO-05	Soil	09/11/95
MHDD79	RA-SE-08	Soil	09/11/95
MHDD80	RA-SE-09	Soil	09/11/95
MHDD83	RA-SE-07	Soil	09/12/95
MHDD86	RA-SE-04	Soil	09/12/95
MHDD88	RA-SE-06	Soil	09/12/95
MHDD89	RA-WSO-03	Soil	09/12/95
MHDD91	RA-SE-03	Soil	09/12/95
MHDD92	RA-SO-01	Soil	09/12/95
MHDD94	RA-SE-02	Soil	09/12/95
MHDD95	RA-WSO-04	Soil	09/12/95
MHDD97	RA-SE-05	Soil	09/12/95
MHDD99	RA-SO-02	Soil	09/12/95

*reviewed
11/6/95
RCC*

DATA QUALITY STATEMENT*

- () Data are ACCEPTABLE according to the Functional Guidelines with no qualifiers (flags) by the reviewer
- (X) Data are acceptable with QUALIFICATIONS noted in review
- () Data are UNACCEPTABLE according to the Functional Guidelines

Telephone/Communication Logs Enclosed? Yes ____ No X

TPO Attention Required? Yes ____ No X

If yes, list the items that require attention:

* Please see Data Qualifier Definitions, attached to the end of this report.

INORGANIC DATA QUALITY ASSURANCE REVIEW

REVIEW NARRATIVE SUMMARY

This data package was reviewed according to the EPA document "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," February 1994.

Case 24008, SDG MHDD72 consisted of 20 soil samples for CLP RAS metals and cyanide analyses.

Water samples MHDA96, MHDD84, and MHDD98 from Case 24008, SDG MHDD70, were designated rinsate blanks and were used to evaluate sample results. Results for these blanks are attached to the end of the report.

The following tables list the data qualifiers added to sample analyses.

SAMPLE ID	ELEMENTS - QUALIFIERS	PROBLEM	REVIEW SECTION
MHDD88	chromium - UJ	Preparation Blank Results	Form 3
MHDD72, MHDD73, MHDD79, MHDD86, MHDD91, MHDD94, MHDD97, MHCQ95, MHCQ96	sodium - UJ		
MHDD72, MHDD73, MHDD75, MHDD76, MHDD79, MHDD80, MHDD83, MHDD88, MHDD92, MHDD94, MHDD95, MHDD99, MHCQ95, MHCQ97	thallium - UJ	Continuing Calibration Blank Results	
MHDD94, MHDD97, MHCQ95, MHCQ96	sodium - UJ		
MHDD83, MHDD94, MHDD95, MHDD99	selenium - J	Negative Continuing Blank Results	
MHCQ95	beryllium - UJ	Rinsate Blank Results	
MHCQ95, MHCQ96, MHDD72, MHDD73, MHDD79, MHDD86, MHDD91, MHDD94, MHDD97	sodium - UJ		

INORGANIC DATA QUALITY ASSURANCE REVIEW

SAMPLE ID	ELEMENTS - QUALIFIERS	PROBLEM	REVIEW SECTION
MHCQ96	thallium, potassium, cadmium - UJ or J	Interference Check Sample	Form 4
MHCQ97	thallium, potassium - J		
MHDD74	antimony - J		
MHDD75	selenium, thallium - J		
MHDD76	thallium, potassium, - J		
MHDD83	selenium, thallium, potassium, - J		
MHDD88	thallium potassium, cadmium, nickel, silver - J		
MHDD89	cadmium - UJ		
MHDD95	selenium, thallium - J		
All Samples	lead, manganese, zinc - J	Matrix Spike Results	Form 5A
All Samples	aluminum, arsenic, calcium, iron, lead, manganese - J	Duplicate Sample Results	Form 6
MHCQ97, MHDD74, MHDD75, MHDD76, MHDD83, MHDD88, MHDD89, MHDD95, MHDD99	sodium - UJ	Negative Sample Results	Additional Comments
MHCQ97, MHDD76, MHDD88	antimony - UJ		
MHDD89	cadmium - UJ		

INORGANIC DATA QUALITY ASSURANCE REVIEW

SOW OLM03.0

RAS INORGANIC DELIVERABLES COMPLETENESS CHECKLIST

P Inorganic Cover Page
P Inorganic Analysis Data Sheets (Form I)
P Initial Calibration and Calibration Verification Results (Form II)
P Continuing Calibration Verification Results (Form II)
P CRDL Standard for ICP & AA (Form II, Part 2)
P Blank Analysis Results (Form III)
P ICP Interference Check Sample Results (Form IV)
P Spiked Sample Results (Form V)
P Post-digest Spiked Sample Analysis (Form V, Part 2)
P Duplicate Sample Results (Form VI)
P Instrument Detection Limits (Form X - Quarterly)
P Laboratory Control Sample results (Form VII)
NA Standard Addition Results (Form VIII)
P ICP Serial Dilution Results (Form IX)
P ICP Interelement Correction Factors (Form XII - Quarterly , or Form XI - Annually)
P ICP Linear Ranges (Form XII - Quarterly)
P Raw Data
 P Samples P Calibration Standards P Blanks P Spikes
 P Duplicates P ICP QC (ICS and Serial Dilution) P LCS
 NA Furnace AA P Mercury Analysis P Cyanide Analysis
P Percent Solids Calculations - Solids Only
P Sample Prep/Digestion Logs (Form XIII)
P Analysis Run Log (Form XIV)
P Traffic Report(s)
P Chain of Custody
P Sample Description
P Case Narrative
P Method References

KEY: P = Provided in original data package, as required by contract
R = Provided as Resubmission
NP = Not provided in original data package or as resubmission
NR = Not required under contract
NA = Not applicable to this data package

Comments: None.

INORGANIC DATA QUALITY ASSURANCE REVIEW

HOLDING TIMES

All CLP-SOW holding times were met.

Yes X No

All technical holding times were met.

Yes X No

Comments: None.

INSTRUMENT CALIBRATION: STANDARDS AND BLANKS

Initial instrument calibrations were performed according to contract requirements.

Yes X No

Comments: None.

The instruments were calibrated daily and each time an analysis run was performed.

Yes X No

Comments: None.

The instruments were calibrated using one blank and the appropriate number of standards.

Yes X No

Comments: None.

FORM 1 - SAMPLE ANALYSIS RESULTS

Sample analyses were entered correctly on the Form I's.

Yes X No

Comments: None.

INORGANIC DATA QUALITY ASSURANCE REVIEW

FORM 2A - INITIAL AND CONTINUING CALIBRATION VERIFICATION

The initial and continuing calibration verification standards (ICV and CCV, respectively) met contract requirements.

Yes X No

Comments: None.

The calibration verification results were within 90-110% recovery for metals, 80-120% for mercury, and 85-115% for cyanide.

Yes X No

Comments: None.

The continuing calibration standards were run at 10% frequency.

Yes X No

Comments: None.

FORM 2B - CRDL STANDARD FOR ICP AND AA

ICP Analysis: Standards (CRI) at 2X the CRDL or the IDL whichever were greater, were analyzed at the beginning and the end of each sample run, or at a minimum of twice per eight hour shift, whichever was more frequent.

Yes X No

Comments: None.

GFAA Analysis: Standards (CRA) at the CRDL or the IDL whichever were greater, were analyzed at the beginning of each sample run.

Yes No N/A X

Comments: None.

The CRI and/or the CRA were analyzed after the ICV.

Yes X No N/A

Comments: None.

INORGANIC DATA QUALITY ASSURANCE REVIEW**FORM 3 - BLANKS**

The initial and continuing calibration blanks (ICB and CCB, respectively) met contract requirements.

Yes X No

Comments: None.

The continuing calibration blanks were run at 10% frequency.

Yes X No

Comments: None.

A laboratory/preparation blank was run at the frequency of one per twenty samples, or per sample delivery group (whichever is more frequent), and for each matrix analyzed.

Yes X No

Comments: None.

All analyzed blanks were free of contamination.

Yes No X

Comments: The following table lists the blanks with contamination, elements present, affected samples, and data qualifiers:

TYPE OF BLANK	ELEMENTS PRESENT; CONCENTRATION	SAMPLES AFFECTED - DATA QUALIFIERS
PBS	chromium; .210 mg/Kg	MHDD88 - UJ
	sodium; 57.91 mg/Kg	MHDD72, MHDD73, MHDD79, MHDD86, MHDD91, MHDD94, MHDD97, MHCQ95, MHCQ96 - UJ
CCB1	thallium; 2.20 µg/L	MHDD72, MHDD73, MHDD75, MHDD76, MHDD79 - UJ
CCB2	thallium; 3.58 µg/L	MHDD72, MHDD73, MHDD75, MHDD76, MHDD79, MHDD80, MHDD83, MHDD88 - UJ
CCB3	selenium; -2.11 µg/L	MHDD83, MHDD94, MHDD95, MHDD99 - J

INORGANIC DATA QUALITY ASSURANCE REVIEW

TYPE OF BLANK	ELEMENTS PRESENT; CONCENTRATION	SAMPLES AFFECTED - DATA QUALIFIERS
CCB4	sodium; 163.8 µg/L	MHDD94, MHDD97, MHCQ95, MHCQ96 - UJ
	thallium; 2.3 µg/L	MHDD92, MHDD94, MHDD95, MHDD99, MHCQ95, MHCQ97 - UJ
CCB5	thallium; 2.1 µg/L	MHCQ97 - UJ
MHDA96 Rinsate 09/13/95	beryllium; 1.0 µg/L	MHCQ95 - UJ
	sodium; 317 µg/L	MHCQ95, MHCQ96 - UJ
MHDD84 Rinsate 09/11/95	sodium; 340 µg/L	MHDD72, MHDD73, MHDD79 - UJ
MHDD98 Rinsate 09/12/95	sodium; 363 µg/L	MHDD86, MHDD91, MHDD94, MHDD97 - UJ

FORM 4 - ICP INTERFERENCE CHECK SAMPLE

The ICP interference check sample (ICS) was run twice per eight hour shift and/or at the beginning and end of each sample set analysis sequence (whichever is more frequent).

Yes X No

Comments: None.

Percent recovery of the analytes in solution ICSAB were within the range of 80-120%.

Yes X No

Comments: None.

INORGANIC DATA QUALITY ASSURANCE REVIEW

The ICSA and ICSAB contained no false positive or false negative results greater than the IDL.

Yes ☐ No ☒

Comments: The following results greater than the IDL were reported for the interference check samples.

ELEMENT	TRUE VALUE	IDL ($\mu\text{g/L}$)	ICSAI ($\mu\text{g/L}$)	ICSAM ($\mu\text{g/L}$)	ICSAF ($\mu\text{g/L}$)
antimony	0	3	3	5	<IDL
barium	0	1	4	4	6
cadmium	0	1	-3	-3	-3
lead	0	1	-5	-3	-4
manganese	0	1	-4	-3	-3
nickel	0	1	2	3	2
potassium	0	834	2905	3021	3353
selenium	0	2	-3	-3	-6
thallium	0	2	5	4	8
zinc	0	1	3	3	3
zinc	0	5	-8	-8	<IDL
manganese	0	2	-6	NA	-4
manganese	0	2	-4	NA	-5
lead	0	1	-3	NA	21*

* Absolute value is greater than the CRDL.

INORGANIC DATA QUALITY ASSURANCE REVIEW

ELEMENT	TRUE VALUE	IDL (µg/L)	ICSABI (µg/L)	ICSABM (µg/L)	ICSABF (µg/L)
antimony	0	3	3.2	<IDL	<IDL
arsenic	0	2	2.4	3.0	2.8
potassium	0	834	3652.1	2158.0	2689.2
sodium	0	142	<IDL	-155.0	-275.1
thallium	0	2	2.8	9.1	3.1

Comments: Qualifications were made for samples with comparable or higher levels of interferents and with analyte concentrations that approximate the levels found in the ICS as false positives or false negatives. Listed below are samples, elements with potential interferences, and qualifiers added to the data.

SAMPLE	ELEMENT	QUALIFIER
MHCQ96	thallium, potassium	J*
	cadmium	UJ
MHCQ97	thallium, potassium	J*
MHDD74	antimony	
MHDD75	selenium, thallium	
MHDD76	thallium, potassium	
MHDD83	selenium, thallium, potassium	J*
MHDD88	thallium, potassium, cadmium, nickel, silver	J*
MHDD89	cadmium	UJ
MHDD95	selenium, thallium	J*

* Elements previously qualified "UJ" for blank results will carry a final "UJ" qualifier.

INORGANIC DATA QUALITY ASSURANCE REVIEW**FORM 5A - MATRIX SPIKE SAMPLE ANALYSIS**

A matrix spike sample was analyzed with every twenty or fewer samples of a similar matrix, or one per sample delivery group (whichever is more frequent).

Yes X No

Comments: None.

Spike recoveries were within the range of 75 - 125% (an exception is granted where the sample concentration is 4 times the spike concentration).

Yes No X

Comments: The following table lists the spike recoveries outside control limits, samples affected, and data qualifiers.

ELEMENT	SPIKE RECOVERY	SAMPLES AFFECTED - QUALIFIERS
lead	66.6%	All Samples - J
manganese	218.4%	
zinc	50.6%	

FORM 5B - POST DIGEST SPIKE RECOVERY

A post-digest spike was performed for those elements that did not meet the specified criteria (exception: Ag, Hg).

Yes X No N/A

Comments: None.

FORM 6 - DUPLICATE SAMPLE ANALYSIS

Duplicate sample analysis was performed with every twenty or fewer samples of a similar matrix, or one per sample delivery group (whichever is more frequent).

Yes X No

Comments: None.

INORGANIC DATA QUALITY ASSURANCE REVIEW

The RPDs were calculated correctly.

Yes X No

Comments: ESAT was unable to confirm the duplicate sample results listed on Form 6 - IN. Recalculated values were within two to four percent of the reported values. Correcting the duplicate results did not affect the number of elements outside the appropriate control limits.

For sample concentrations >5 times the CRDL, RPDs were within $\pm 20\%$ (limits of $\pm 35\%$ apply for soil/sediments/tailings samples).

Yes No X N/A

Comments: Several elements were outside control limits for relative percent difference in the duplicate sample analysis. The following table lists duplicate results outside control limits, samples affected, and data qualifiers.

ELEMENT	RPD	SAMPLES AFFECTED - QUALIFIERS
aluminum	39.5%	All Samples - J
iron	43.9%	
lead*	151.4%	
manganese*	51.9%	

* No additional qualifiers were added to elements previously qualified for matrix spike results.

For sample concentrations <5 times the CRDL, duplicate analysis results were within the control window of \pm CRDL (2X CRDL for soils).

Yes No X

Comments: Two elements were outside the control window of two times the CRDL in the duplicate sample analysis. The following table lists duplicate results outside control limits, samples affected, and data qualifiers.

INORGANIC DATA QUALITY ASSURANCE REVIEW

ELEMENT	2x CRDL LIMITS mg/Kg	REPORTED DIFFERENCE (mg/Kg)	SAMPLES AFFECTED - QUALIFIERS
arsenic	5.64	18.3	All Samples - J
calcium	2820	22,860	

GFAA QC

GFAA analyses was not performed for this SDG.

FORM 7 - LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) was prepared and analyzed with every twenty or fewer samples of a similar matrix, or one per sample delivery group (whichever is more frequent). An aqueous LCS is not required for mercury.

Yes X No

Comments: None.

All results were within the control limits.

Yes X No

Comments: None.

FORM 8 - STANDARD ADDITION RESULTS

Results from graphite furnace standard additions were correctly entered on Form I and Form VIII.

Yes No N/A X

Comments: None.

FORM 9 - ICP QC

A serial dilution was performed for ICP analysis with every twenty or fewer samples of a similar matrix, or one per sample delivery group, whichever is more frequent.

Yes X No

Comments: None.

INORGANIC DATA QUALITY ASSURANCE REVIEW

The serial dilution was without interference problems as defined by the functional guidelines.

Yes X No

Comments: None.

FORM 10 - QUARTERLY INSTRUMENT DETECTION LIMITS (IDL)

IDL's were provided for all elements on the target analyte list.

Yes X No

Comments: None.

Reported IDL's met contract requirements.

Yes X No

Comments: None.

CYANIDE INSTRUMENT DETECTION LIMITS (IDL)

An IDL for cyanide was provided in the raw data.

Yes X No N/A

Comments: None.

The reported cyanide IDL met contract requirements.

Yes X No N/A

Comments: None.

FORM 11 - INTERELEMENT CORRECTION FACTORS FOR ICP

Interelement corrections for ICP were reported and met contract requirements.

Yes X No

Comments: None.

INORGANIC DATA QUALITY ASSURANCE REVIEW**FORM 12 - ICP LINEAR RANGES**

ICP linear ranges were reported and met contract requirements.

Yes X No

Comments: None.

FORM 13 - PREPARATION LOG

Information on the preparation of samples for analysis was reported on Form XIII.

Yes X No

Comment: None.

FORM 14 - ANALYSIS RUN LOG

A Form XIV with the required information was filled out for each analysis run in the data package.

Yes X No

Comments: None.

Additional Comments or Problems/Resolutions (not addressed above).

Listed below are samples with negative results greater than the IDL and qualifier.

SAMPLE	ELEMENT	NEGATIVE CONCENTRATION (mg/Kg)	QUALIFIER
MHCQ97	sodium	-248	UJ
	antimony	-5.87	
MHDD74	sodium	-784	
MHDD75		-114	
MHDD76		-407	
	antimony	-4.03	

INORGANIC DATA QUALITY ASSURANCE REVIEW

SAMPLE	ELEMENT	NEGATIVE CONCENTRATION (mg/Kg)	QUALIFIER
MHDD83	sodium	-399	UJ
MHDD88		-7280	
	antimony	-44.1	
MHDD89	sodium	-296	
	cadmium	-1.44	
MHDD95	sodium	-319	
MHDD99		-432	

INORGANIC DATA QUALITY ASSURANCE REVIEW

REGION VIII

DATA QUALIFIER DEFINITIONS

For the purpose of Data Validation, the following code letters and associated definitions are provided for use by the data validator to summarize the data quality. Use of additional qualifiers should be carefully considered. Definitions for all qualifiers used should be provided with each report.

GENERAL QUALIFIERS for use with INORGANIC DATA

- R** - Reported value is "rejected". Resampling or reanalysis may be necessary to verify the presence or absence of the compound.
- J** - The associated numerical value is an estimated quantity because the Quality Control criteria were not met.
- UJ** - The reported amount is estimated because Quality Control criteria were not met. Element was not detected.

TARGET SHEET
EPA REGION VIII
SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 374981

SITE NAME: RICO ARGENTINE/RICO POND

DOCUMENT DATE: 11/06/1995

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☒ TYPES OF DOCUMENTS NOT TO BE SCANNED
(Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

INORGANIC ANALYSES DATA SHEETS
